

# National Bureau of Standards

## Certificate of Analysis

### Standard Reference Material 1570

#### Trace Elements in Spinach

This Standard Reference Material is intended primarily for calibrating instrumentation and evaluating the reliability of analytical methods for the determination of major, minor, and trace elements in agricultural food products and other botanical materials.

**Certified Values of Constituent Elements:** The certified values for the constituent elements are shown in Table 1. They are based on results obtained either by reference methods of known accuracy or by two or more independent, reliable analytical methods. Noncertified values, which are given for information only, appear in Table 2.

All values are based on a minimum sample size of 300 mg of the material dried as indicated under "Instructions for Drying."

**Notice and Warnings to Users:**

**Expiration of Certification:** This certification will be invalid after 5 years from the date of shipping. Should it be invalidated before then, purchasers will be notified by NBS.

**Stability:** The material should be kept in its original bottle and stored at temperatures between 10-30 °C. It should not be exposed to intense sources of radiation, including ultraviolet lamps or sunlight. Ideally, the bottle should be kept in a desiccator in the dark at the temperature indicated.

**Use:** The bottle should be shaken well before each use. A minimum sample of 300 mg of the *dried* material (see Instructions for Drying) should be used for any analytical determination to be related to the certified values of this certificate.

The overall direction and coordination of the technical measurements leading to this certificate were performed under the chairmanship of H. L. Rook.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. Alvarez.

Washington, D.C. 20234  
September 1, 1976

J. Paul Cali, Chief  
Office of Standard Reference Materials

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**Additional Information on Analyses:** This Standard Reference Material contains siliceous material, which is an integral part of the sample. The analyses reported in Tables 1 and 2 were performed on the entire sample. Therefore, dissolution procedures should be capable of complete dissolution of the sample but should not result in losses of volatile elements, such as arsenic and mercury.

**Preparation of Material:** The spinach for this Standard Reference Material was obtained and freeze-dried by Oregon Freeze Dry Foods, Inc., Albany, Oregon. At NBS, the lyophilized material was sieved through a polypropylene sieve having openings of 0.25 mm (equivalent to a U.S. Series 60 standard sieve) and blended to provide a more homogeneous material. It was then radiation sterilized with 2.5 megarads of cobalt-60 radiation at Neutron Products, Inc., Dickerson, Md.

**Homogeneity Assessment:** Material homogeneity was evaluated by determining ten of the certified elements, Al, Fe, Mn, Zn, Rb, Cu, Cr, As, U, and Hg, on samples of 300 mg or less taken at various locations of the freeze-dried bulk material. The other certified elements, K, Ca, P, Sr, Pb, and Th were determined using sample weights not exceeding one gram. The uncertainties for the concentrations given in Table I include these results.

**Instructions for Drying:** Samples of this Standard Reference Material *must* be dried before weighing by either of the following procedures:

1. Drying in air in an oven at 85 °C for 2 hours.
2. Lyophilization using a cold trap at or below -50 °C at a pressure *not greater* than 30 Pa (0.2 mm Hg) for 24 hours.

**NOTE:** Drying either in an oven at 105 °C or in a vacuum oven at 25 °C causes large losses of volatiles other than water and should *not* be used.

#### Analytical Methods Used and Analysts

##### Analytical Methods

- A. Atomic absorption spectroscopy
- B. Isotope dilution mass spectrometry
- C. Isotope dilution spark source mass spectrometry
- D. Kjeldahl method for nitrogen
- E. Neutron activation
- F. Nuclear track technique
- G. Optical emission spectroscopy
- H. Spectrophotometry
- I. Polarography

##### Analysts

##### Analytical Chemistry Division, National Bureau of Standards

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Table 1. Certified Values of Constituent Elements<sup>a</sup>

Major and Minor Constituents

<u>Element</u>	<u>Content Wt. Percent</u>
Potassium	3.56 ± 0.03
Calcium	1.35 ± 0.03
Phosphorus	0.55 ± 0.02

Trace Constituents

<u>Element</u>	<u>Content μg/g</u>	<u>Element</u>	<u>Content μg/g</u>
Aluminum	870 ± 50	Chromium	4.6 ± 0.3
Iron	550 ± 20	Lead	1.2 ± 0.2
Manganese	165 ± 6	Arsenic	0.15 ± 0.05
Strontium	87 ± 2	Thorium	0.12 ± 0.03
Zinc	50 ± 2	Uranium	0.046 ± 0.009
Rubidium	12.1 ± 0.2	Mercury	0.030 ± 0.005
Copper	12 ± 2		

<sup>a</sup> Analytical values are based on the "dry-weight" of material (See Instructions for Drying).

The uncertainties of the values of the constituents shown in Table 1 include allowances for material inhomogeneity, method imprecision, and an estimate of possible biases of the analytical methods used.

Table 2. Non-certified Values for Constituent Elements<sup>a</sup>

**NOTE:** The values shown in this table are not certified because they are not based on the results of either a reference method of known accuracy or of two or more independent reliable methods. These values are included for information only.

Major Constituent

<u>Element</u>	<u>Content Wt. Percent</u>
Nitrogen	(5.9)

Trace Constituents

<u>Element</u>	<u>Content μg/g</u>	<u>Element</u>	<u>Content μg/g</u>
Bromine	(54)	Lanthanum	(0.37)
Boron	(30)	Scandium	(0.16)
Nickel	(6)	Antimony	(0.04)
Cadmium <sup>b</sup>	(1.5)	Thallium	(0.03)
Cobalt	(1.5)	Europium	(0.02)

<sup>a</sup> Analytical values are based on the "dry-weight" of material (See Instructions for Drying).

<sup>b</sup> Cadmium was found to be not sufficiently homogeneous for certification.